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U.S. DEPARTMENT OF COMMERCE/Technology Administration
National Institute of Standards and Technology

Standard Reference Materials:

Polystyrene Films for Calibrating the Wavelength Scale of Infrared Spectrophotometers — SRM 1921

Devinder Gupta, Lan Wang, Leonard M. Hanssen,
Jack J. Hsia, and Raju U. Datla

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Standard Reference Materials:
Polystyrene Films for Calibrating the Wavelength Scale of Infrared Spectrophotometers - SRM 1921

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ABSTRACT

Standard Reference Material (SRM) 1921 is a matte finish polystyrene film and is intended for use in calibrating the wavelength scale of spectrophotometers in the infrared (IR) spectral region from 545.48 cm^{-1} to 3082.19 cm^{-1} (18.3325 μm to 3.2445 μm). Thirteen absorption peak positions are certified using a center-of-gravity technique. The expanded uncertainty values associated with these peak values are between 0.06 cm^{-1} and 0.66 cm^{-1}, except for 1.84 cm^{-1} at 2850.13 cm^{-1} and 12.29 cm^{-1} at 545.48 cm^{-1}. This publication describes the IR spectrophotometer, instrument calibration, SRM material, measurement procedure, calibration of polystyrene films, and uncertainty determination.

Keywords: Fourier transform infrared spectrophotometer; infrared wavelength standard; polystyrene film; standard reference material; wavelength scale; wavelength standard.

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1. Introduction

Fourier transform infrared (FT-IR) spectrophotometers are extensively used for chemical, optical and astronomical studies and have gained popularity over dispersive instruments because of high resolution and speed. Although the Michelson interferometer was invented in the nineteenth century, it only became commercially available much later as a Fourier transform spectrometer with the advent of lasers and high speed computers [1].

The ubiquitous use of FT-IR instruments has led to the need to establish a wavelength scale compatible with its high resolution. From the beginning of infrared spectroscopy, it was realized that a stable material for the calibration of spectrometers was required. A number of materials were suggested including polystyrene, 1,2,4-chlorobenzene, toluene, and other compounds. Among these materials polystyrene proved the most useful and is often used for the calibration of infrared spectrometers. Plyler and coworkers [2] at the National Bureau of Standards (now, the National Institute of Standards and Technology {NIST}) made a careful evaluation of the wavelengths of the absorption bands in polystyrene. It was noted that a number of absorption bands which were not resolved with a sodium chloride prism, were resolved with a lithium fluoride prism, thereby indicating that the wavelength designation for some of the absorption bands would depend upon the resolution of the spectrometer. The reference wavelengths provided by them are often employed for the calibration of prism and grating instruments. Hannah and Farnum [3] have investigated the effects of interference and resolution on the absorption bands of polystyrene using a dispersive infrared spectrometer. The standard practice for measuring the performance of dispersive infrared spectrometers is described in the American Society for Testing and Materials (ASTM) standard E932-89 [4]. A similar standard has also been prepared for FT-IR instruments by ASTM : Standard E1421-91 [5].

The present work has been undertaken with the objective of developing polystyrene film as a Standard Reference Material (SRM) which would provide reference wavenumber and wavelength values for the calibration of wavenumber and wavelength scales of infrared (IR) spectrophotometers. This is accomplished by first calibrating a NIST FT-IR spectrometer using water vapor and carbon dioxide absorption lines. Even in well purged or evacuated FT-IR instruments, water vapor and carbon dioxide remain in the residual air in the sample compartment of the instrument and provide absorption lines with adequate intensities. The performance of the instrument is evaluated by comparing the measured absorption bands to the standard bands available in the literature [6]. After the instrument was calibrated, measurements were performed on a large number of polystyrene films. This has led to the development of a polystyrene film SRM 1921. For IR spectrophotometers with relatively low resolution and with less well purged sample compartments having high partial pressure, the polystyrene film is more useful for wavelength calibration, since water vapor and carbon dioxide absorption bands cannot be used in this situation.

2. Description of the FT-IR spectrophotometer

The instrument employed is a Bomem DA-3.02 [7], which is a FT-IR spectrophotometer having a maximum optical retardation of 50 cm corresponding to a maximum resolution of 0.02 cm^{-1}. A water cooled SiC source is used for the mid-IR region. The aperture size can be varied from 10 mm to 0.5 mm in diameter. The system employs a HeNe laser for position measurement and mirror dynamic alignment. The detectors employed for the mid-IR region are a photoconductive mercury cadmium telluride (MCT) type and a pyroelectric type, but the more sensitive liquid-nitrogen-cooled MCT detector is generally preferred. A coated KBr beam splitter has been used for the present experiment whose spectral range is 500 cm^{-1} to 5000 cm^{-1}. The throughput of the system is matched from the source to the detector for efficient operation of the instrument at various wavelengths and resolutions.

The instrument can be operated in either a purge or vacuum mode . The interferometer, the sample compartment, and the detector housing can be evacuated without disturbing the alignment. The pressure in the sample compartment has been maintained at less than one torr to avoid possible pressure-shift effects on the absorption lines of water vapor and carbon dioxide [8,9]. The instrument room temperature was maintained near 22 °C and the humidity ranged from 30 % to 50 % during the measurements.

3. FT-IR spectrometer calibrations

3.1 Measurement of water vapor and carbon dioxide absorption bands

High resolution spectra of water vapor and carbon dioxide have been obtained by operating the instrument in vacuum mode. The pressure inside the sample compartment was maintained at 50 Pa (0.4 torr). The measurements were performed in the spectral region of 500 cm^{-1} to 2500 cm^{-1} using apodized resolutions of 0.074 cm^{-1} and 0.020 cm^{-1}. Sufficiently strong absorption lines for water vapor have appeared in the spectral region from 1400 cm^{-1} to 2000 cm^{-1}, whereas the spectrum has shown carbon dioxide with absorption bands in the region from 2300 cm^{-1} to 2400 cm^{-1}. The strengths of the various bands depend upon the partial pressure and temperature of these gases in the instrument.

The water molecule has an intense $\sqrt{}_2$ band covering a broad spectral region centered at 1596 cm^{-1}. A knowledge of this band and the surrounding ones is of interest for various purposes, particularly for the accurate calibration of the spectral scale. Guelachvili [8] has provided a high resolution spectrum of the $\sqrt{}_2$ band and reported two thousands absorption bands from 1066 cm^{-1} to 2296 cm^{-1}. We observed a large number of water vapor bands in the spectral region from 1350 cm^{-1} to 2100 cm^{-1} and a smaller number in the spectral region from 1100 cm^{-1} to 1350 cm^{-1} [9]. Since it is not possible to tabulate all the bands in the present work, we have chosen a representative group of 25 bands between 1416 cm^{-1} and 2041 cm^{-1}. A comparison is provided between the observed absorption bands and the standard bands in Table 1. Two sets of measurements made within the span of three years are presented. These were taken with an aperture diameter of 1.5 mm. Another set of measurements was also made

with an aperture diameter of 1 mm but the results did not vary significantly with the change in aperture size. In Table 1 the maximum difference between the observed and standard absorption bands is 0.015 cm^{-1}.

Carbon dioxide, being a simple and common molecule, has been the subject of many spectroscopic investigations [9,10,11]. Guelachvili [12] has reported low pressure Doppler limited absorption spectra around 2300 cm^{-1} and observed about seven hundred bands. We have also observed a large number of absorption bands. A representative group of 22 bands has been selected and compared with the standard values in Table 2. The results of the measurements of carbon dioxide bands are similar to those obtained for water vapor bands. Carbon dioxide bands are also not affected by a change of aperture size from 1.5 mm to 1 mm. Two sets of measurements taken of carbon dioxide bands within a span of about 3 years have maximum difference of 0.015 cm^{-1} when compared to the standard values.

A linear least squares fit of the difference values observed for the standard absorption bands of water vapor and carbon dioxide was performed and used to correct the wavelength scale of the FT-IR instrument. This resulted in a correction of 0.01 cm^{-1} to the certified peak wavenumber values of the ten largest wavenumber peaks.

3.2 Effect of instrument resolution

Interferograms of water vapor bands in the range from 1850 cm^{-1} to 1950 cm^{-1} were measured with a resolution of 0.074 cm^{-1}. These interferograms were used to compute spectra with low resolution. The results were compared with water vapor spectra which were measured with the same low resolution.

A comparison of the computed and measured peak positions of water bands is shown in Tables 3 and 4. A spectrum with a resolution of 0.5 cm^{-1} and a two-sigma standard deviation of 0.020 cm^{-1} was computed from a measured interferogram with a resolution of 0.074 cm^{-1}. Another spectrum with a resolution of 0.5 cm^{-1} was measured directly with a two-sigma standard deviation of 0.024 cm^{-1}. Table 3 shows that the maximum difference between the two sets of data is 0.003 cm^{-1}.

A spectrum with a resolution of 1.0 cm^{-1} and a two-sigma standard deviation of 0.042 cm^{-1} was computed from a measured interferogram with a resolution of 0.074 cm^{-1}. Another spectrum with a resolution of 1.0 cm^{-1} was measured directly with a two-sigma standard deviation of 0.040 cm^{-1}. Table 4 shows that the maximum difference between the two sets of data is 0.079 cm^{-1}.

A comparison of spectra with resolutions of 0.074 cm^{-1}, 0.5 cm^{-1}, and 1.0 cm^{-1} indicates that the differences are smaller than the combined standard deviation. These data indicate that it is possible to use the highest possible resolution to calibrate the wavelength scale and to use a suitable low resolution to measure different samples.

4. Description of the material/polystyrene film

A single roll of matte-finish polystyrene film, having the trade name of Trycite #DWF-6001 manufactured by the Dow Chemical Company [7], was donated by the Coblentz Society, an affiliation of the Society for Applied Spectroscopy. Two thousand five hundred samples were cut from different sections of the roll. Each sample was assigned a number representing the position of the sample on the roll. The thickness of the film is approximately 38 μm and is nearly uniform throughout the entire roll. SRM 1921 has a 25 mm diameter exposed area, centered 38 mm from the bottom of a cardboard holder which is 5 cm (w) x 11 cm (h) x 2 mm (t) in dimension.

5. Measurement procedure for polystyrene film characterization

The polystyrene film transmittance measurements are performed over the spectral range from 500 cm^{-1} to 3500 cm^{-1} with an unapodized resolution of 0.5 cm^{-1}. The Hamming (also known as Happ-Genzel function) [13] apodization function is used. The source aperture diameter sizes are 1 mm for the MCT measurements and 10 mm for the DTGS measurements. The measurements are made in vacuum mode, with a pressure of less than 50 Pa (0.4 Torr). After a suitable time for the instrument to equilibrate, measurements are begun.

First, a reference spectrum is obtained. It is a Fourier transformed single beam spectrum of the empty sample holder. Subsequently, a spectrum of the sample at normal incidence is obtained, which is normalized by the reference spectrum to provide its transmittance spectrum. In each run, twelve samples of polystyrene film have been measured in groups of four after a single reference measurement. This process is repeated six times for each group of twelve polystyrene samples. These measurements are made with a remotely controlled sixteen-position sample changer.

6. Calibration of polystyrene films

6.1 Selection between vacuum and purge

The infrared transmittance spectrum of a polystyrene film obtained in the spectral region from 500 cm^{-1} to 3500 cm^{-1} is shown in Fig. 1. Twenty-eight absorption lines were selected for investigation. Their nominal wavenumbers are similar to the ones in Table 5 with an additional band at 1492 cm^{-1}. Transmittance measurements of polystyrene film samples were made both in purge and in vacuum modes. Positions of the transmittance minima ("peaks") were obtained by using a center-of-gravity (CG) technique which is described in detail in Appendix B. Plotted in Fig. 2(a) are the two sigma standard deviation values as a function of wavenumber for the absorption bands, for measurements taken in vacuum mode (Vacuum, CG) and those taken in purge mode (Purge, CG). The absolute difference of measurements in these two modes (indicated by the symbol ❑ for |Vac Peak- Purge Peak|) is shown in Fig. 2(b). Even though these peak differences are less than the two sigma standard deviations, it is evident from Fig. 2(a) that the two sigma standard deviation values for vacuum

mode are slightly less than those for purge mode. All further measurements were performed in vacuum.

6.2 Selection of the peak wavenumber value determination method

After a transmittance spectrum has been obtained, the peak positions (in wavenumbers) of the absorption bands have to be determined. The positions of the peaks can be determined generally by using either a minimum-find technique or a center-of-gravity technique [14]. In the minimum-find technique the derivative of the spectrum is obtained and a threshold limit is set up. The differentiated values are searched to determine the peaks that have excursions both above and below the threshold level. In the case of the center-of-gravity technique, an initial selection of peaks is made based on a threshold limit and then the peak position is determined as described in detail in Appendix B. If the peak is symmetric, the peak position at the minimum (transmittance) is determined. For an asymmetric peak, the results will deviate from this value dependent on the degree of asymmetry and the number of points around the center used in the determination of the peak. In Fig. 3(a), as a function of wavenumber values, the two sigma standard deviations for the peak positions determined by this center-of-gravity technique (CG, Vacuum) are, in general, one order less than those determined by the minimum-find technique (Min Find, Vacuum). For the center-of-gravity technique 29 wavenumber values (28 bands as shown in Table 5 plus 1492 cm^{-1}) are used. And for the minimum-find technique 21 wavenumber values (20 bands as shown in Table 5 plus 1492 cm^{-1}) are used. The absolute difference (indicated by the symbol ❑ for |CG Peak - Min Peak|) between the peak positions determined by the center-of-gravity and the minimum-find techniques shown in Fig. 3(b), are larger than their two sigma standard deviations. The number of wavenumber values are the same as that in Fig. 3(a). Therefore, this particular center-of-gravity method was chosen for all further investigations to determine the peak positions.

6.3 Selection of the reference peaks

Out of those absorption peaks observed in the spectrum of a polystyrene film (Fig. 1), thirteen peaks have been chosen for further calibration. The criterion for the selection of these peaks are based on the associated uncertainty and the intensity of the peak. For a selected peak, the value of the uncertainty associated must be small. Furthermore, the peak should neither be so strong that its minimum approaches the zero percent transmittance nor should it be so shallow that it is subjected to interference effects.

For the selection of these peaks, 12 polystyrene film samples were measured six times each. Twenty-eight average peak positions are presented in Table 5. The peaks at approximate positions of 624 cm^{-1}, 1183 cm^{-1}, 1325 cm^{-1}, 1368 cm^{-1}, 1542 cm^{-1}, 1667 cm^{-1}, 1746 cm^{-1}, 1873 cm^{-1}, and 2343 cm^{-1} have large two-standard deviations [15,16]. Their values range from 0.544 cm^{-1} to 4.3 cm^{-1}. Whereas the peaks near 699 cm^{-1}, 757 cm^{-1}, 1450 cm^{-1}, 1492 cm^{-1}, 2921 cm^{-1} and 3026 cm^{-1} are the strongest ones and are likely to approach the zero percent transmittance, the peaks having approximate positions of 965 cm^{-1}, 1003 cm^{-1}, 1804 cm^{-1}, and 1945 cm^{-1} are generally weak and likely be affected by the interference. Thus, 13 peak positions were selected for further calibration.

6.4 Calibration of the polystyrene films

The certified wavenumbers and wavelengths values of these thirteen peaks are obtained from the data on 60 representative polystyrene films. In Table 6, four wavenumbers are starred to indicate that for those wavenumbers the difference between the peak values using a center-of-gravity technique and a minimum-find technique is less than 0.1 cm^{-1} (Refer to Fig. 3b). Details of sample selection and grand mean peak value calculation are presented in Section 7.2. The peak near 545 cm^{-1} is included in the reference wavenumbers to cover a larger range from 500 cm^{-1} to 3500 cm^{-1}, although it has a large uncertainty of 12.29 cm^{-1}. Furthermore, the peak at 3026 cm^{-1} is also included even though it is highly intense.

The calibration measurements were made with a resolution of 0.5 cm^{-1}. The ambient temperature of the instrument was maintained near 22 °C and the range of the humidity was from 30 % to 50 % during the measurements. The calibration measurements were made under vacuum at pressures of 50 Pa (0.4 Torr). The incident beam was normal to and focused onto the sample with an f-number of 4. A center-of-gravity method was used to find the peak wavenumber values from a spctrum of transmittance vs wavenumbers.

The wavelength values in Table 6 are vacuum values (where n=1, the index of refraction). For measurements of these polystyrene films made under nitrogen gas or dry air purge, the wavenumber and wavelength values need to be adjusted due to the index of refraction of air or purge gas (n=1.00026 for dry nitrogen gas at atmospheric pressure and T=298 K) [17,18]. The measured wavenumber values should be divided by 1.00026 and the wavelength values should be multiplied by 1.00026 to compare to the standard vacuum values [18]. However, for instruments which give vacuum wavenumber and wavelength values, no adjustment for the index of refraction of air or purge gas should be performed.

7. Uncertainty determination

7.1 Instrument uncertainties

The correction to the wavelength scale of the FT-IR from the instrument calibrations using water vapor and carbon dioxide absorption bands with standard values resulted in corrections of 0.01 cm^{-1} and 0.00 cm^{-1} with an uncertainty of 0.005 cm^{-1}.

7.2 Sample, run, and measurement uncertainties

After the selection of the 13 peak bands, additional measurements were conducted on polystyrene film samples to determine the representative wavenumber values as well as the sample-to-sample, run-to-run, and measurement-to-measurement variations. A total of 60 samples were measured in six different runs. The procedure adopted was random sample selection for a multivariate analysis. In the first four runs, 48 samples roughly evenly distributed between sample numbers 9 and 315 were selected and measured. Each run had 12 samples. The samples were placed in a carousel, and the carousel was cycled through the

measurement process six times. Run 5 was measured in the same manner as the first four, except that the 12 samples were selected between sample numbers 9 and 2500, i.e., out of the entire batch of samples. In run 6 the same samples as in run 5 were measured, but with a different detector. In order to provide each detector the same weight when computing the grand mean for each peak, the mean for run 6 and the mean from the first five runs were averaged.

A multivariate analysis of variance on data from these runs was also undertaken and the results are presented in Table 7 together with the mean wavenumber values. These results indicate that the sample, run, and measurement uncertainties are statistically significant. A considerable variability from sample to sample indicates that the material is not homogenous throughout the entire roll. Furthermore, a significant difference has been observed from run to run and the variability between them may be related to the repeatability of the instrument since the runs have been measured at different periods of time. The measurement uncertainty is reasonably small, thereby indicating that the measurement procedure adopted is quite reliable.

7.3 Detector uncertainty

In run 6 the samples were measured with a DTGS detector while in runs 1 to 5 all samples were measured with a MCT detector. Table 7 shows the uncertainties of the absorption peaks due to different detectors. The variability may be due to the following factors. The DTGS detector requires higher intensity source corresponding to the large source aperture; hence the sample may be at a higher temperature; the MCT detector has a greater non-linearity; the large source aperture image on the sample may result in less interference. On the other hand, water vapor and CO_2 bands are very sharp and were not influenced by measurements using different detectors.

7.4 Total uncertainty

In Table 7, the combined standard uncertainty, u_c, is the root sum square of the standard uncertainties from detector, sample, run, and measurement. The combined standard uncertainty has an estimate of degree of freedom and this was used to determine the coverage factor, k [15,16]. The expanded uncertainty [15,16], U, is the product of the combined standard uncertainty and the coverage factor which was obtained at a 95% level of confidence. The effects of intensity variation, beam geometry, and peak-determination method on the peak wavenumber values are briefly discussed in Appendix A.

8. SRM 1921 certificate

The certificate for SRM 1921 is presented in Appendix B. The following items are included: certified wavenumber and wavelength values with associated uncertainties; description of the measurement conditions; instructions for storage, handling, and use of SRM 1921.

9. Summary

A Fourier transform infrared spectrophotometer has been calibrated using the absorption bands of water vapor and carbon dioxide. The spectral transmittance of a representative group of polystyrene film samples has been measured in vacuum. The positions of absorption peaks in the region of 545.48 cm^{-1} to 3082.19 cm^{-1} (18.3325 μm to 3.2445 μm) were obtained using a center-of-gravity method. Thirteen wavelength values were selected and certified along with their associated uncertainties. The measurement uncertainties caused by detector, sample, and run to run statistical variation are included. Different peak-determination methods are compared.

10. Acknowledgment

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Table 1
Observed and Standard Peak Wavenumbers of 25 Water Vapor Bands

(1) (cm^{-1})	(2) (cm^{-1})	(3) standard value [6] (cm^{-1})	(1)-(3) (cm^{-1})	(2)-(3) (cm^{-1})
1416.080		1416.08664	-0.007	
1417.247		1417.25367	-0.007	
1417.492		1417.49879	-0.007	
1418.926		1418.93334	-0.007	
1419.311		1419.31757	-0.007	
1419.502		1419.50841	-0.006	
1423.697		1423.70452	-0.008	
1424.123		1424.13032	-0.007	
	1505.599	1505.60461		-0.006
1601.201		1601.20825	-0.007	
1602.877		1602.88452	-0.008	
1603.313		1603.32020	-0.007	
1792.651	1792.652	1792.65937	-0.008	-0.007
1795.793		1795.80232	-0.008	
1796.144		1796.13289	-0.008	
1799.608		1799.61605	-0.008	
1802.472	1802.473	1802.48013	-0.008	-0.007
1807.696		1807.70376	-0.008	
1810.621	1810.623	1810.62861	-0.008	-0.006
1942.508	1942.509	1942.51651	-0.009	-0.008
1942.757		1942.76573	-0.009	
1945.332		1945.34064	-0.009	
1946.357		1946.36477	-0.008	
2018.323		2018.33791	-0.015	
2041.279		2041.28876	-0.010	

(1) 0.074 cm^{-1} resolution;
(2) 0.02 cm^{-1} resolution measured three years later than (1)

Table 2
Observed and Standard Peak Wavenumbers of 22 CO_2 Bands

(1) (cm^{-1})	(2) (cm^{-1})	(3) standard value [6] (cm^{-1})	(1)-(3) (cm^{-1})	(2)-(3) (cm^{-1})
2330.548	2330.550	2330.55754	-0.010	-0.008
2332.359	2332.360	2332.36934	-0.010	-0.009
2334.146	2334.146	2334.15678	-0.011	-0.010
2335.910	2335.909	2335.91984	-0.010	-0.011
2337.648	2337.650	2337.65849	-0.010	-0.008
2339.362	2339.364	2339.37269	-0.011	-0.007
2341.053	2341.053	2341.06244	-0.009	-0.005
2342.738	2342.717	2342.72769	-0.010	-0.011
2344.358	2344.359	2344.36842	-0.010	-0.009
2346.000	2345.976	2345.98461	-0.015	-0.009
2351.437	2351.437	2351.44763	-0.010	-0.011
2354.423	2354.423	2354.43380	-0.011	-0.011
2355.880	2355.883	2355.88983	-0.010	-0.007
2357.311	2357.311	2357.32133	-0.010	-0.010
2358.721	2358.721	2358.73605	-0.015	-0.015
2361.457	2361.459	2361.46638	-0.009	-0.007
2362.789	2362.790	2362.79850	-0.010	-0.009
2364.095	2364.096	2364.10576	-0.011	-0.010
2365.377	2365.380	2365.37958	-0.003	-0.000
2366.636	2366.637	2366.64560	-0.010	-0.009
2367.870	2367.869	2367.87815	-0.008	-0.009
2369.076	2369.075	2369.08574	-0.010	-0.011

(1) 0.074 cm^{-1} resolution;
(2) 0.02 cm^{-1} resolution measured 3 years later than (1)

Table 3
Peak Wavenumber of Water Vapor Bands
and the effect of resolution (0.5 cm^{-1})

(1) (cm^{-1})	2σ	(2) (cm^{-1})	2σ	(1)-(2)
1866.373	0.018	1866.370	0.042	0.003
1869.332	0.012	1869.335	0.012	-0.003
1889.560	0.016	1889.561	0.014	-0.001
1895.187	0.020	1895.190	0.030	-0.003
1907.940	0.042	1907.937	0.028	0.003
1909.951	0.032	1909.948	0.034	0.003
1918.014	0.008	1918.014	0.006	0.000
1923.149	0.018	1923.152	0.018	0.003

(1) Computed for 0.5 cm^{-1} resolution
(2) Measured at 0.5 cm^{-1} resolution

Table 4

Peak Wavenumber of Water Vapor Bands and the effect of resolution (1 cm^{-1})

(1) cm^{-1}	2σ	(2) cm^{-1}	2σ	(1)-(2)
1866.389	0.030	1866.363	0.024	0.026
1869.354	0.050	1869.380	0.016	-0.026
1889.558	0.038	1889.550	0.086	-0.008
1895.183	0.044	1895.177	0.018	0.006
1908.007	0.064	1907.928	0.072	0.079
1910.016	0.064	1909.939	0.062	0.077
1918.032	0.018	1917.987	0.012	0.045
1923.144	0.026	1923.140	0.034	0.004

(1) Computed for 1.0 cm^{-1} resolution
(2) Measured at 1.0 cm^{-1} resolution

Table 5
Peak Wavenumber of Twenty-Eight Measured Absorption Bands of Polystyrene Films (12 films with 6 measurements on each film)

peak (cm^{-1})	2σ	peak (cm^{-1})	2σ
546.940*	0.240	1542.162*	0.644
623.799*	0.800	1583.147	0.010
699.448	0.034	1601.366	0.016
756.580	0.022	1745.598*	0.896
841.989	0.154	1803.825	0.516
906.785	0.004	1872.821*	0.602
965.714	0.322	1945.241	0.514
1028.318	0.078	2343.085*	4.300
1069.097	0.120	2849.994	0.012
1154.613	0.004	2920.935	0.042
1183.007	1.056	3001.424	0.014
1325.065*	2.560	3026.375	0.016
1368.527*	0.544	3060.020	0.012
1449.675	0.038	3082.226	0.008

* These bands are not included for Min. Find, Vacuum in Fig. 3a and are not included for peak difference in Fig. 3b.

Table 6
Thirteen Certified Peak Wavenumber and Wavelength Values
(in vacuum)

peak Wavenumber (cm^{-1})	expanded uncertainty	peak wavelength (μm)	expanded uncertainty
545.48	12.29	18.3325	0.4129
842.08	0.49	11.8754	0.0070
906.82	0.66	11.0275	0.0080
1028.35	0.27	9.7243	0.0026
1069.20	0.46	9.3528	0.0040
1154.64*	0.54	8.6607*	0.0041
1583.13*	0.06	6.3166*	0.0002
1601.35*	0.07	6.2447*	0.0003
2850.13	1.84	3.5086	0.0023
3001.40	0.12	3.3318	0.0001
3026.42	0.61	3.3042	0.0007
3060.03*	0.14	3.2680*	0.0002
3082.19	0.12	3.2445	0.0001

* Peak values less sensitive to peak determination method.

Table 7
Detector, Sample, Run, and Measurement Uncertainties

no.	peak (cm^{-1})	<-----uncertainties (σ)(cm^{-1})-----> detector	sample	run	measurement	combined u_C	**expanded uncertainty $U = k\ u_C$**	coverage factor k
1	**545.48**	1.345	0.414	0.049	0.027	1.409	**12.287**	8.72
2	**842.08**	0.112	0.199	0.000	0.018	0.228	**0.494**	2.17
3	**906.82**	0.053	0.006	0.013	0.002	0.055	**0.662**	12.05
4	**1028.35**	0.066	0.080	0.017	0.008	0.106	**0.271**	2.57
5	**1069.20**	0.104	0.185	0.000	0.012	0.212	**0.456**	2.15
6	**1154.64**	0.038	0.000	0.018	0.001	0.042	**0.540**	12.83
7	**1583.13**	0.000	0.012	0.023	0.001	0.025	**0.059**	2.32
8	**1601.35**	0.000	0.018	0.024	0.002	0.030	**0.066**	2.21
9	**2850.13**	0.134	0.000	0.046	0.004	0.142	**1.843**	13.01
10	**3001.40**	0.000	0.012	0.047	0.000	0.048	**0.121**	2.49
11	**3026.42**	0.034	0.014	0.049	0.002	0.061	**0.609**	9.93
12	**3060.03**	0.000	0.009	0.053	0.002	0.054	**0.136**	2.53
13	**3082.19**	0.000	0.011	0.047	0.000	0.048	**0.121**	2.49

Table 8
Effect of Intensity Variation on the Peak Wavenumber of the Absorption Bands of a Polystyrene Film
(Ten measurements for each band)

	(1) (cm $^{-1}$)	(2σ) (cm $^{-1}$)	(2) (cm $^{-1}$)	(2σ) (cm $^{-1}$)	(2)-(1) (cm $^{-1}$)
1	544.824	0.024	545.345	0.036	+0.521
2	842.328	0.006	842.341	0.008	+0.013
3	906.800	0.004	906.874	0.006	+0.074
4	1028.282	0.004	1028.352	0.004	+0.070
5	1068.976	0.006	1069.006	0.006	+0.030
6	1154.597	0.004	1154.645	0.002	+0.048
7	1583.074	0.006	1583.137	0.003	+0.063
8	1601.279	0.007	1601.351	0.004	+0.072
9	2849.923	0.020	2849.908	0.020	-0.015
10	3001.334	0.010	3001.327	0.010	-0.007
11	3026.266	0.014	3026.301	0.016	+0.035
12	3059.889	0.012	3059.870	0.008	-0.019
13	3082.068	0.012	3082.105	0.003	+0.037

(1) With screen
(2) Without screen

Table 9
Effect of Beam Geometry on the Peak Wavenumber of the Absorption Bands of a Polystyrene Film
(Ten measurements for each band)

	(1)	(2σ)	(2)	(2σ)	(2)-(1)
1	546.047	0.042	544.824	0.025	-1.223
2	841.483	0.006	842.328	0.008	+0.845
3	906.908	0.004	906.800	0.004	-0.108
4	1028.365	0.039	1028.282	0.004	-0.083
5	1069.203	0.006	1068.976	0.006	-0.227
6	1154.676	0.004	1154.597	0.004	-0.080
7	1583.152	0.003	1583.074	0.006	-0.078
8	1601.359	0.008	1601.279	0.007	-0.080
9	2849.920	0.018	2849.923	0.020	+0.003
10	3001.304	0.008	3001.334	0.010	+0.030
11	3026.325	0.018	3026.266	0.014	-0.059
12	3059.869	0.016	3059.899	0.012	+0.030
13	3082.080	0.010	3082.068	0.012	-0.012

(1) 45° tilt
(2) No tilt

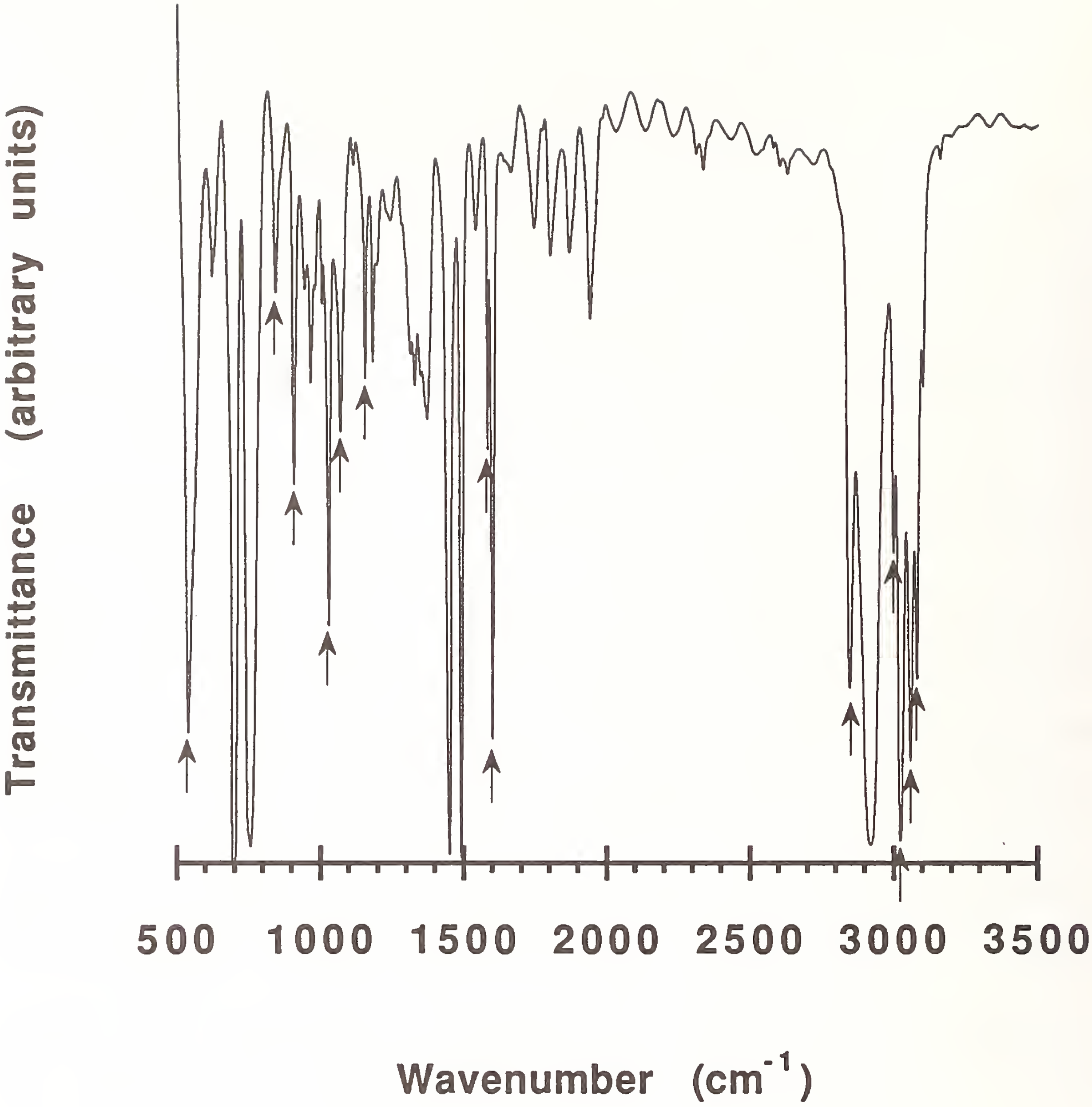

Figure 1. Spectrum of polystyrene film showing locations of certified absorption peaks.

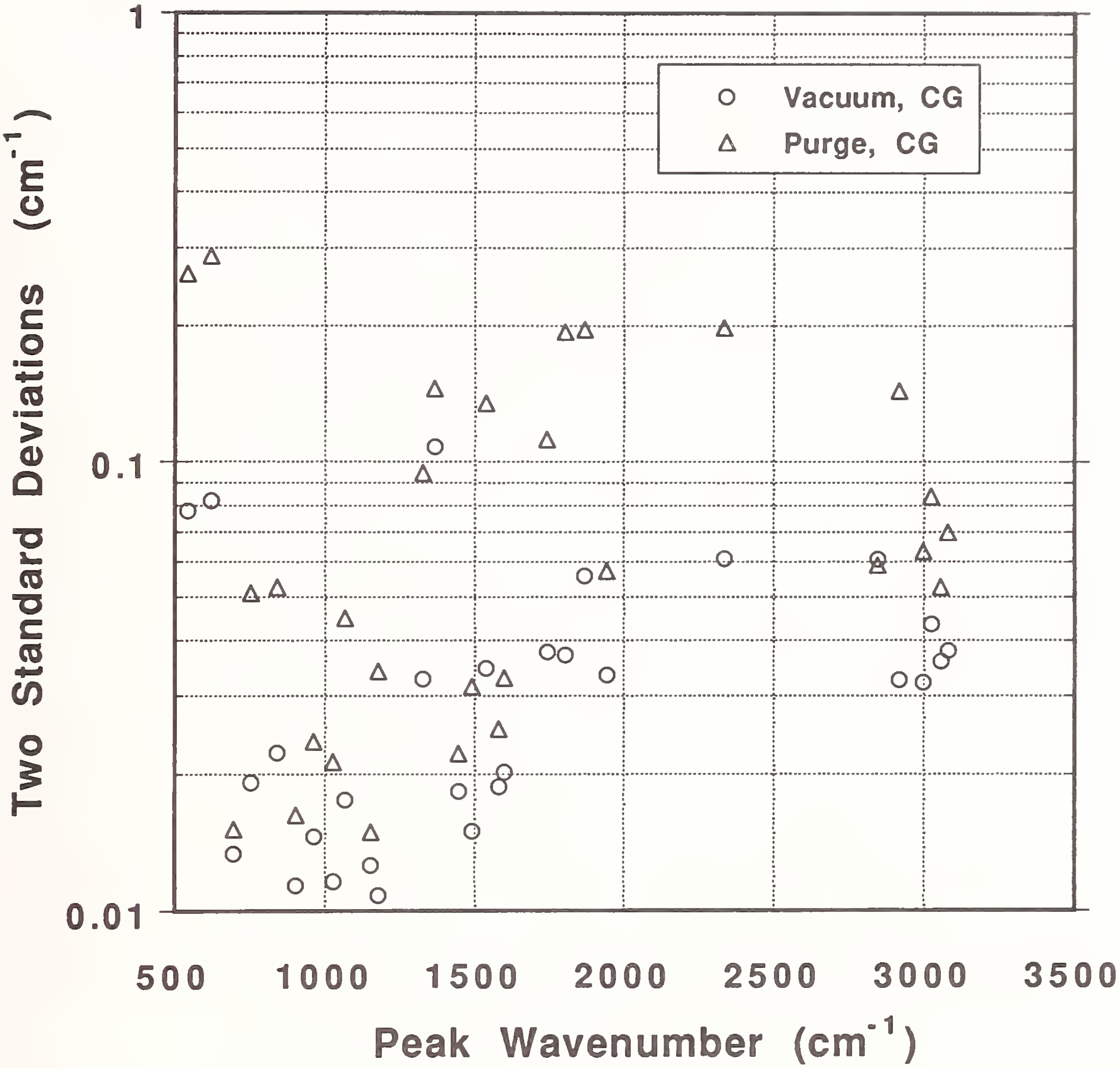

Figure 2a. Comparison of peak positions for polystyrene absorption bands, made under vacuum and purge conditions: two standard deviation values.

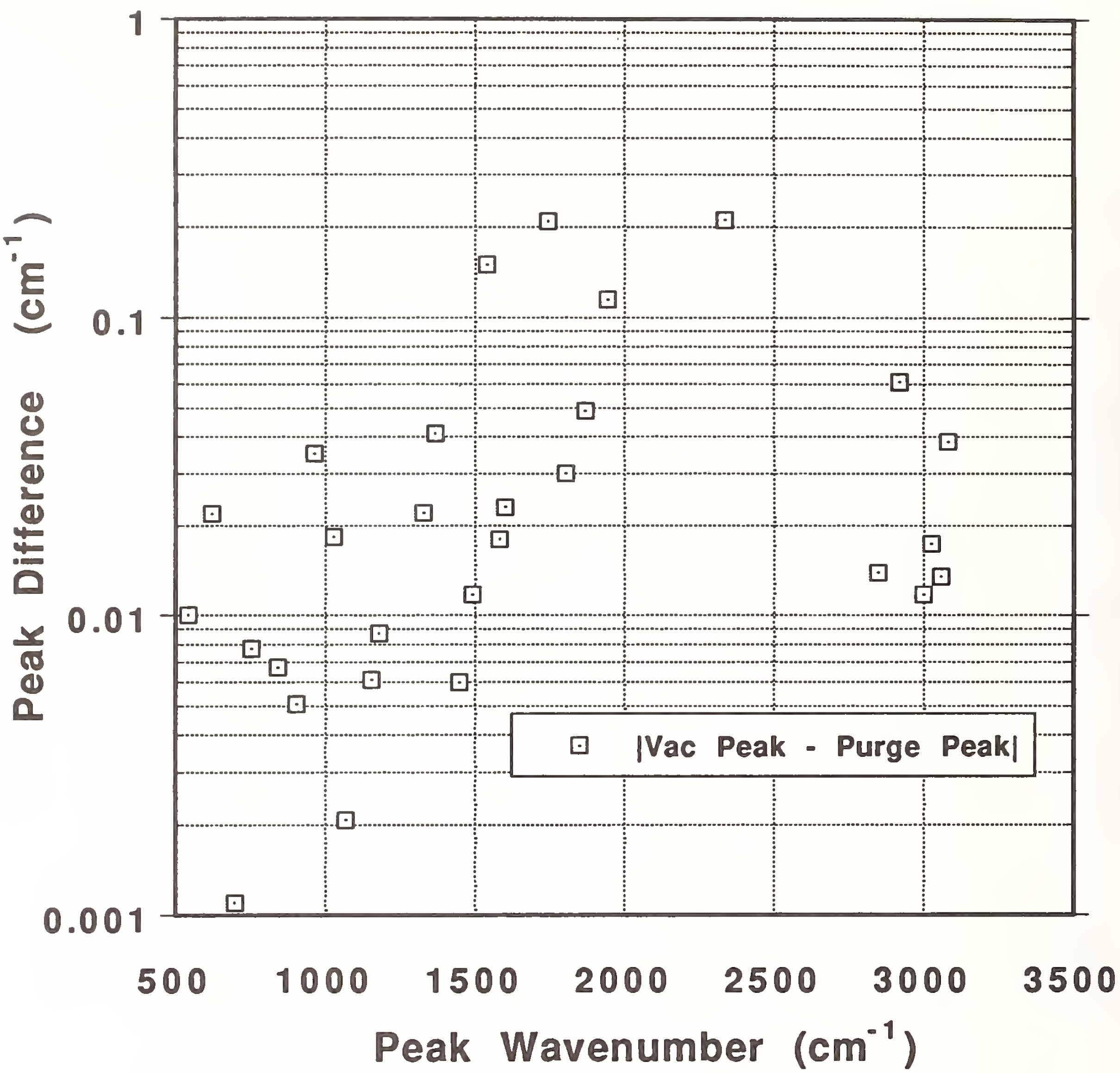

Figure 2b. Comparison of peak positions for polystyrene absorption bands, made under vacuum and purge conditions: peak position differences.

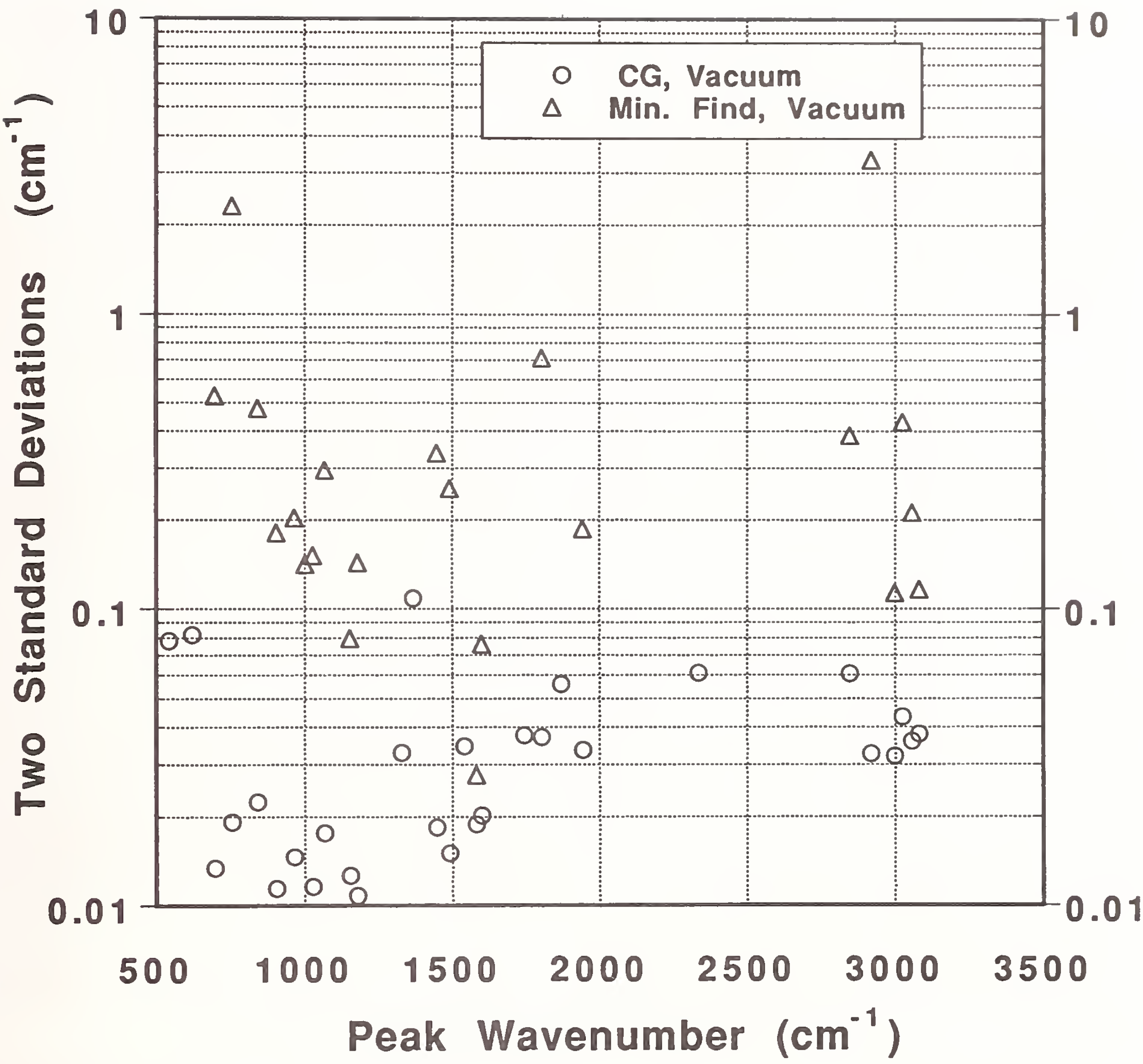

Figure 3a. Comparison of peak positions for polystyrene absorption bands, as determined by center-of-gravity and minimum-find techniques: two standard deviation values.

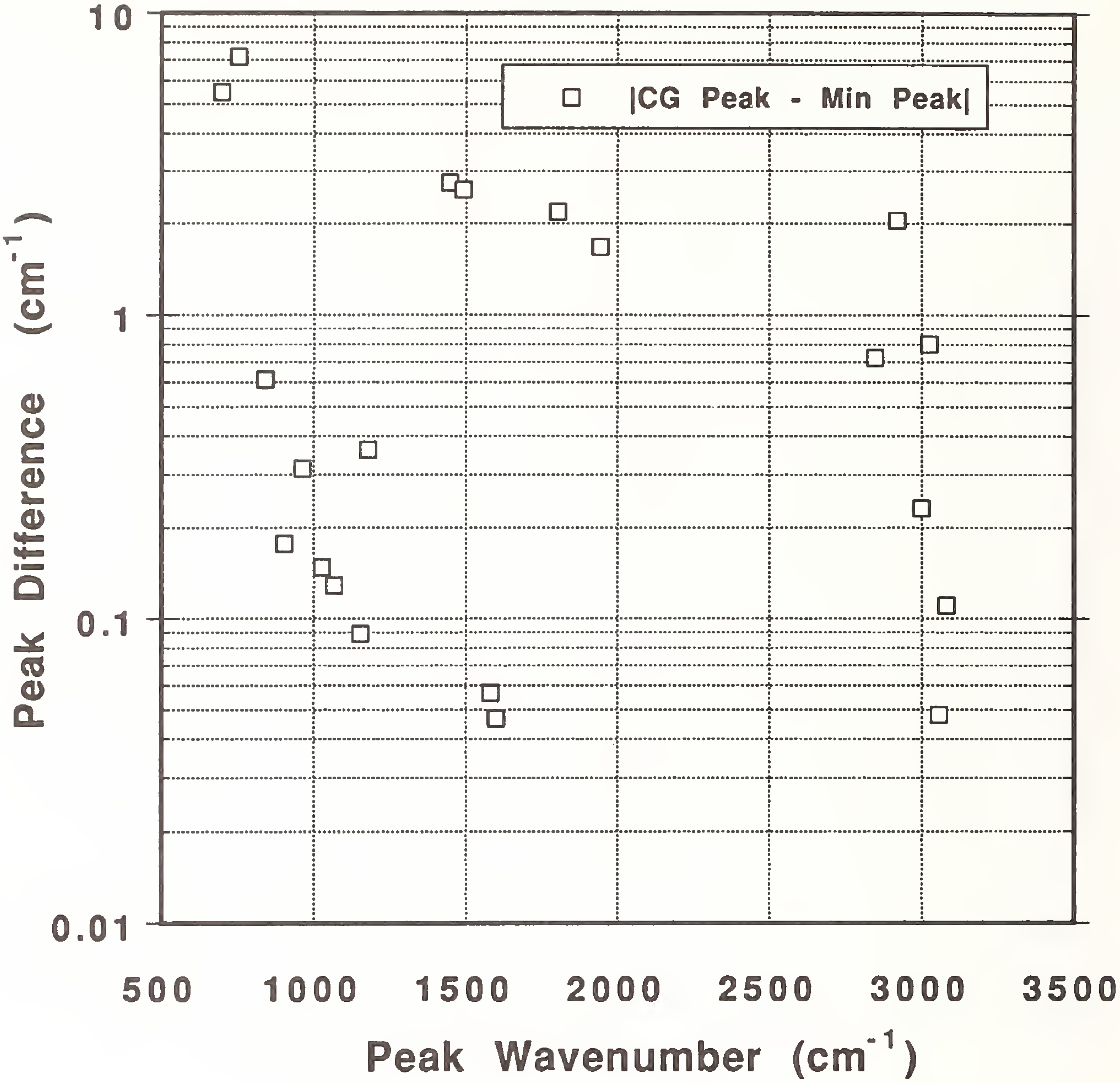

Figure 3b. Comparison of peak positions for polystyrene absorption bands, as determined by center-of-gravity and minimum-find techniques: peak position differences.

Appendix A. Effects of several parameters

A.1 effect of intensity variation

The absorption peaks of a polystyrene film were obtained with and without using a screen in the spectrometer. Table 8 indicates that there are statistically significant differences. The screen was used to reduce the intensity on the detector in a neutral fashion to test for peak value dependence on source intensity. The variation may be related to the MCT detector nonlinearity.

A.2 Effect of beam geometry

A polystyrene film has been measured with normal incidence and with 45° incidence. This was done to reveal any dependence on beam geometry and/or path length in the polystyrene film. Even with this large incident angle, the absorption-peak difference between measurements with 45° and with normal angle exceeds the expanded uncertainty at only three peaks as shown in Table 9.

A.3 Effect of peak-determination method

The exact peak positions of a polystyrene film may be different when using different peak wavenumber determination methods to obtain the peak wavenumbers. Fig. 3(b) shows the peak difference obtained by a center-of-gravity (CG) technique and by a minimum-find (Min Find) technique in vacuum (indicated by the symbol ❑ for {CG Peak - Min Peak}). Most of the differences are larger than the two standard deviations of both techniques shown in Fig. 3(a).

Appendix B. SRM 1921 certificate

(see next six pages)

National Institute of Standards & Technology

Certificate

Standard Reference Material® 1921

Infrared Transmission Wavelength Standard

This Standard Reference Material (SRM) is intended for use in calibrating the wavelength scale of spectrophotometers in the infrared (IR) spectral region from 3.2 μm to 18 μm (555 cm^{-1} to 3125 cm^{-1}). SRM 1921 is a matte finish polystyrene film approximately 38 μm thick with a 25 mm diameter exposed area, centered 38 mm from the bottom of a cardboard holder which is 5 cm (w) x 11 cm (h) x 2 mm (t) in size. A unit of SRM 1921 consists of five (5) polystyrene cards.

Certified Wavelength Values: The spectral transmittance of a representative group of samples of polystyrene film was measured in vacuum. The positions of absorption peaks in the range of 3.2 μm to 18 μm were obtained using a center of gravity method. Thirteen of these peak positions were selected for certification based on experimental and statistical analysis results. The wavelength values of these peaks are certified and are shown in Table 1 along with their associated uncertainties. The corresponding peak wavenumber values are listed in Table 2. See the section "Peak Wavenumber and Wavelength Determination" for details. To aid the user in distinguishing among the peaks during measurement, a spectrum is shown in Figure 1, with arrows identifying the certified peaks. For peak wavelength and wavenumber values measured in air or under purge conditions see the section "Correction for Air/Nitrogen Purge".

Measurement Conditions: The calibration measurements were made using a Bomem DA-3.02 Fourier transform spectrophotometer. The instrument room temperature was maintained near 22 °C and the humidity ranged from 30% to 50% during the measurements. Calibration measurements were made under vacuum level pressures of 50 Pa (0.4 Torr). Details of the measurements and data analysis can be found in Reference [1].

Storage and Handling: When not in use, SRM 1921 should always be kept in its accompanying protective cover. For storage, it is advisable to keep the SRM in a desiccator cabinet when available. The SRM should always be handled with care; the exposed film surface should never be touched by fingers or any other objects. Dust may be removed by blowing with clean, dry air.

Expiration of Certification: The polystyrene films have been measured over a period of approximately one year, without significant change of any wavelength positions. The measured samples will be monitored over time, and in the event that the certification becomes invalid, users will be notified by NIST. Because of the finite measurement period, this certificate is valid for 3 years from the date of shipment from NIST.

The technical measurements leading to certification were performed by D. Gupta, L.M. Hanssen, and L. Wang of the NIST Radiometric Physics Division. The overall direction and coordination of the technical measurements leading to certification were performed under the supervision of J.J. Hsia, R. Datla, and L.M. Hanssen of the NIST Radiometric Physics Division.

Statistical consultation was provided by S.B. Schiller of the NIST Statistical Engineering Division.

The technical and support aspects involved in the revision of this certificate were coordinated through the Standard Reference Materials Program by J.C. Colbert.

Gaithersburg, MD 20899
April 7, 1995
(Revision of certificate dated 5-19-94)

Thomas E. Gills, Chief
Standard Reference Materials Program

Source of Material: The polystyrene film used in SRM 1921 has been taken from a single roll and was manufactured by the Dow Chemical Company under the trade name Trycite[1], #DWF-6001. The polystyrene film was donated by the Coblentz Society.

INSTRUCTIONS FOR USE

Calibration Measurements: Prior to calibration with SRM 1921, the spectrophotometer should be set up under the following conditions: 1) the instrumental resolution should be set at 0.5 cm^{-1} (if this is not achievable, the resolution should be set at the highest, (i.e. smallest value in cm^{-1}) achievable by the instrument); 2) the spot size on the sample should be set to the maximum possible, yet not greater than that required to maintain measurement resolution and not greater than that at which the wavenumber shift error becomes significant [2] and not greater than that at which the detector becomes significantly non-linear [3,4]; 3) the sample chamber should be closed and purged or evacuated for a suitable time, for the instrument to reach pressure and temperature equilibrium; 4) the calibration procedure should begin with a "reference" measurement with no sample in the sample chamber; 5) SRM 1921 should be placed in the standard sample position, and a "sample" measurement should be made. The ratio of "sample" to "reference" spectra is the transmittance. This process (steps 3 to 5) should be repeated in sequence at least six (6) times. The resulting transmittance spectra should be analyzed for peak position as described below.

Peak Wavenumber and Wavelength Determination: The method used to determine the peak wavenumber (ν) and wavelength (λ) values of SRM 1921 is the center of gravity technique [5]. This procedure is performed on the transmittance spectra using wavenumber values only. The wavenumber value is defined as the number of waves per unit length (cm). Refer to Figure 2 for the following peak determination procedure. First, the bounds of an initial wavenumber range (ν_{min} to ν_{max}) enclosing the peak are determined; they are the wavenumber values at which transmittance relative maxima occur on either side of the peak. Next, the differences between the transmittance values at each bound and the transmittance at the absorption peak

$$\Delta T(\nu_{max}) = [T(\nu_{max})-T(\nu_{peak})] \text{ and } \Delta T(\nu_{min}) = [T(\nu_{min})-T(\nu_{peak})]$$

are determined. Then half the value of the smaller of $\Delta T(\nu_{max})$ and $\Delta T(\nu_{min})$, $\Delta T_o/2$ is determined. The final wavenumber range (ν_1 to ν_2) to be used in the center of gravity calculation is that between the wavenumber values where the transmittance values on either side of the peak equal the minimum transmittance value plus $\Delta T_o/2$.

$$\nu_{1,2}(T=T(\nu_{peak}) + \Delta T_o/2)$$

A center of gravity calculation [6] on this region should be performed to obtain the peak wavenumber values to compare to the certified values. If another peak wavenumber determination method is used, a comparison with the certified values may <u>not</u> be valid. Four peak wavenumber values, noted in Tables 1 and 2, were found to be less sensitive to the technique used to derive them (see Reference 1). Peak values determined by other techniques may become available in the future. Wavelength values can be obtained from the wavenumber values through the relationship $\lambda\nu=1$, where λ is the wavelength in cm.

Correction for Air/Nitrogen Purge: The calibration measurements were performed in vacuum. Hence the wavelength values in Table 1 are vacuum values (where n=1, the index of refraction). For measurements of SRM 1921 made under nitrogen gas or air purge, the wavenumber and wavelength values need to be adjusted[2] due to the index of refraction of the air or purge gas (n=1.00026 for dry nitrogen gas at atmospheric pressure and (T=298 K)) [7,8]. The measured wavenumber values should be divided by 1.00026 and the wavelength values should be multiplied by 1.00026 to compare to the standard values.

---

[1] The use of a trade name on this certificate is for identification only and does not imply endorsement of the product by the National Institute of Standards and Technology.

[2] For instruments which give vacuum wavenumber and wavelength values, no adjustment for the index of refraction of air or purge gas should be performed.

Corrections to Instrument Wavenumber Scale: The resulting N ($N \geq 6$) values for each peak at wavenumber, ν, should be averaged to obtain a single "peak wavenumber value" (laboratory mean, $\bar{y}_\nu$), and the standard deviation of the values, s_ν, should be calculated. In order to determine whether the laboratory measurements are biased relative to SRM 1921, calculate the absolute difference, Δ_ν, between the laboratory mean, $\bar{y}_\nu$ and the certified value, C_ν:

$$\Delta_\nu = | \bar{y}_\nu - C_\nu |$$

The uncertainty associated with this difference is:

$$\Delta_c = (t_{N-1}0.95)\ s/\sqrt{N} + U,\ [9]$$

where $t_{N-1}0.95$ is the critical value from the student's distribution with N-1 degrees of freedom for a two-sided 95% confidence interval and U [10,11] is the uncertainty from the certificate. For example, if N=6, t_5 0.95 = 2.571. If $\Delta_\nu > \Delta_c$, then the difference is greater than can be explained by chance, and the wavenumber scale of the instrument should be corrected to the SRM. If this is the case, generally a linear least squares fit of Δ_ν to ν for the thirteen peaks should provide a sufficient correction to the spectrophotometer scale. However, if $\Delta_\nu \leq \Delta_c$, the wavenumber scale of the spectrophotometer is accurate and correction is not advised.

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Table. 1 Certified Peak Wavelength Values (in vacuum)

Peak Wavelength (μm)	Expanded Uncertainty, U[a]
18.3325	0.4129
11.8754	0.0070
11.0275	0.0080
9.7243	0.0026
9.3528	0.0040
8.6607*	0.0041
6.3166*	0.0002
6.2447*	0.0003
3.5086	0.0023
3.3318	0.0001
3.3042	0.0007
3.2680*	0.0002
3.2445	0.0001

Table. 2 Certified Peak Wavenumber Values (in vacuum)

Peak Wavenumber (cm^{-1})	Expanded Uncertainty, U[a]
545.48	12.29
842.08	0.49
906.82	0.66
1028.35	0.27
1069.20	0.46
1154.64*	0.54
1583.13*	0.06
1601.35*	0.07
2850.13	1.84
3001.40	0.12
3026.42	0.61
3060.03*	0.14
3082.19	0.12

[a]The expanded uncertainty, a NIST associated uncertainty of the certified peak value, was calculated according to "The Guide to Expression of Uncertainty in Measurement" [10, 11]. It is the product of a coverage factor, at the 95% level of confidence, and the combined standard uncertainty. The combined standard uncertainty is the root sum of squares of components of uncertainty due to detector, sample, run, and measurement.

*Peak values less sensitive to peak determination method [1].

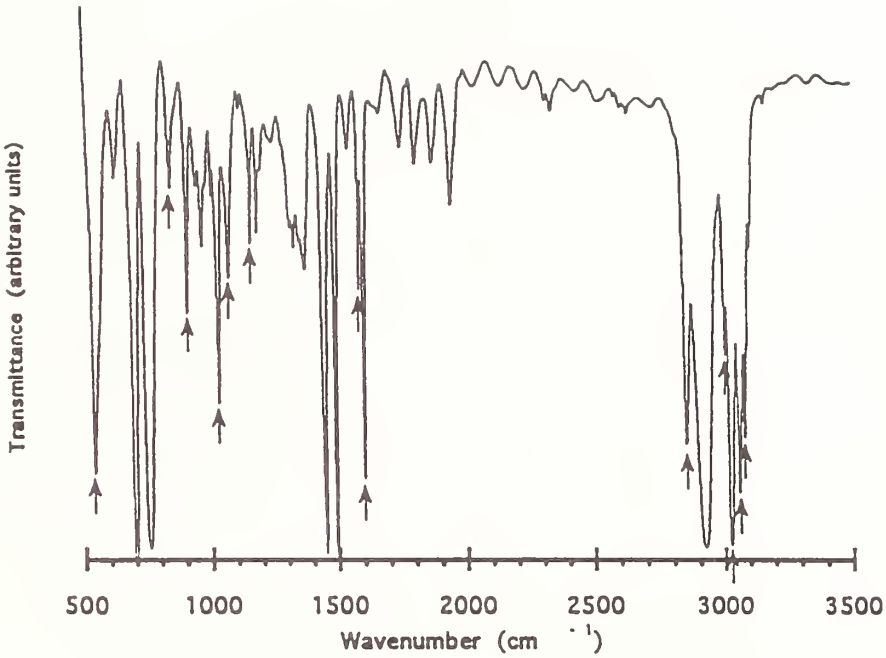

Figure 1. Spectrum of polystyrene film showing locations of certified peaks.

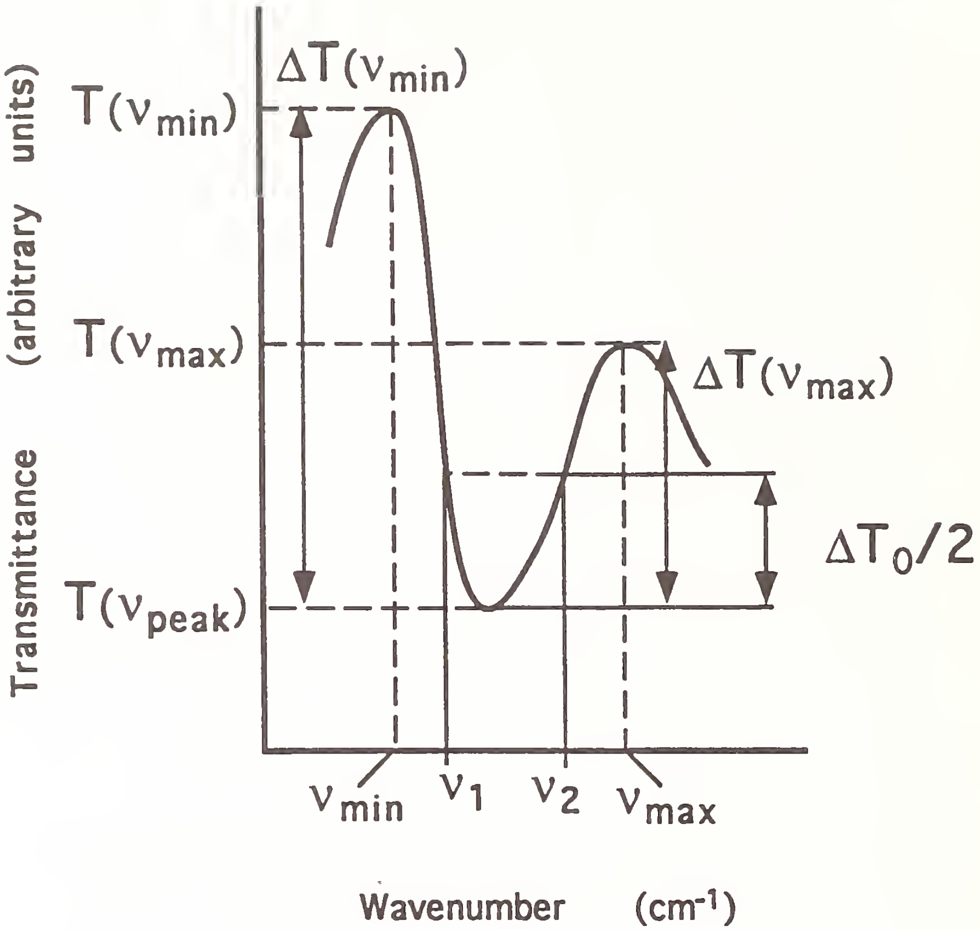

Figure 2. Diagram indicating parameters used in the peak wavenumber determination method (see text for details).

NIST *Technical Publications*

Periodical

Journal of Research of the National Institute of Standards and Technology—Reports NIST research and development in those disciplines of the physical and engineering sciences in which the Institute is active. These include physics, chemistry, engineering, mathematics, and computer sciences. Papers cover a broad range of subjects, with major emphasis on measurement methodology and the basic technology underlying standardization. Also included from time to time are survey articles on topics closely related to the Institute's technical and scientific programs. Issued six times a year.

Nonperiodicals

Monographs—Major contributions to the technical literature on various subjects related to the Institute's scientific and technical activities.

Handbooks—Recommended codes of engineering and industrial practice (including safety codes) developed in cooperation with interested industries, professional organizations, and regulatory bodies.

Special Publications—Include proceedings of conferences sponsored by NIST, NIST annual reports, and other special publications appropriate to this grouping such as wall charts, pocket cards, and bibliographies.

National Standard Reference Data Series—Provides quantitative data on the physical and chemical properties of materials, compiled from the world's literature and critically evaluated. Developed under a worldwide program coordinated by NIST under the authority of the National Standard Data Act (Public Law 90-396). NOTE: The Journal of Physical and Chemical Reference Data (JPCRD) is published bimonthly for NIST by the American Chemical Society (ACS) and the American Institute of Physics (AIP). Subscriptions, reprints, and supplements are available from ACS, 1155 Sixteenth St., NW, Washington, DC 20056.

Building Science Series—Disseminates technical information developed at the Institute on building materials, components, systems, and whole structures. The series presents research results, test methods, and performance criteria related to the structural and environmental functions and the durability and safety characteristics of building elements and systems.

Technical Notes—Studies or reports which are complete in themselves but restrictive in their treatment of a subject. Analogous to monographs but not so comprehensive in scope or definitive in treatment of the subject area. Often serve as a vehicle for final reports of work performed at NIST under the sponsorship of other government agencies.

Voluntary Product Standards—Developed under procedures published by the Department of Commerce in Part 10, Title 15, of the Code of Federal Regulations. The standards establish nationally recognized requirements for products, and provide all concerned interests with a basis for common understanding of the characteristics of the products. NIST administers this program in support of the efforts of private-sector standardizing organizations.

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NIST Interagency Reports (NISTIR)—A special series of interim or final reports on work performed by NIST for outside sponsors (both government and nongovernment). In general, initial distribution is handled by the sponsor; public distribution is by the National Technical Information Service, Springfield, VA 22161, in paper copy or microfiche form.

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